USEPA REGION 9 LABORATORY RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 380 PURGEABLE HYDROCARBONS BY GC FID

Revision 8 Effective Date: January 15, 2014

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1 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the procedures used to determine total petroleum hydrocarbons in the gasoline range (TPH-GRO) by gas chromatography (GC) in water and solid matrices. This SOP is based on procedures from EPA SW 846 Method 5030C Revision 3, May 2003; Method 5035A Draft Revision 1, July 2002; and Method 8015C Revision 3, February 2007. Deviations from reference methods are described in Appendix A.

Analytes and quantitation limits are provided by matrix in Appendix B.

2 METHOD SUMMARY

An inert gas is bubbled through a portion of an aqueous sample (or methanol extract from solid samples). Volatile organic compounds are vaporized and swept through a sorbent column where they are adsorbed. The sorbent column is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column. A temperature program is used in the gas chromatograph to separate the organic compounds followed by detection using a flame ionization detector (FID).

TPH-DRO is quantitated by determining the retention times of 2-methylpentane and 1,2,4-trimethylbenzene and using these markers to establish the retention time range of the gasoline. The area sum response of the sample over this retention time range is compared to the area sum response of gasoline standards analyzed under the same conditions. Probable identification of gasoline in samples is done by comparing the chromatographic pattern generated by analysis of the sample to the chromatographic pattern generated by analysis of gasoline standard analyzed under the same conditions. The identification of TPH as a specific fuel type may be complicated by environmental processes such as evaporation, biodegradation, or the presence of more than one fuel type.

3 DEFINITIONS

A list of terms and definitions specific to this procedure appears below. For terms and acronyms in general use at the EPA Region 9 Laboratory refer to Appendix A of the Laboratory Quality Assurance Plan.

<u>Storage Blank (SB)</u> – An aliquot of reagent water stored with samples in the sample storage refrigerator. The storage blank indicates whether contamination may have occurred during sample storage.

<u>Gasoline Range Organics (GRO)</u> Hydrocarbons that elute in the GC retention time range from 2-methylpentane and to 1,2,4-trimethylbenzene, as described in Section 8.2.1. This corresponds approximately to a carbon range of C_6 - C_{10} .

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4 SAFETY & HEALTH

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation must be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN at I:\MSDS IMAGES for additional information.

Safety precautions must be taken when handling solutions and samples. Protective clothing including laboratory coats, safety glasses, and gloves must always be worn. Contact lenses must not be worn. If solutions come into contact with your eyes, flush with water continuously for 15 minutes. If solutions come in contact with your skin, wash thoroughly with soap and water. ESAT personnel should contact the Group Leader or Health and Safety and Environmental Compliance Task Manager and EPA staff should see the Team Leader or the Laboratory Safety, Health and Environmental Compliance Manager to determine if additional treatment is required. Refer to the Material Safety Data Sheets located in the library and the LAN at I:\MSDS IMAGES for additional information.

4.1.1 Methanol

Methanol is the primary solvent used for the preparation of standards and for soil sample extraction in these procedures. Methanol is harmful if inhaled and may be fatal or cause blindness if ingested. Symptoms of overexposure via inhalation are drowsiness and intoxication, headache, visual disturbances leading to blindness, coughing, shortness of breath, collapse, and death at high concentrations. Skin contact may result in absorption producing toxic effects. Repeated skin contact may cause burning, itching, redness, blisters or dermatitis. Eye contact can cause burning, watering, redness, and swelling. High vapor concentration will result in similar symptoms in the eyes. Medical attention must be sought whenever symptoms of inhalation or ingestion are observed as many effects are delayed due to the slow rate of metabolism.

Methanol is classified as a flammable solvent and must be handled accordingly.

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Use methanol in a laboratory fume hood with appropriate personal protective equipment (laboratory coat, nitrile gloves, and safety glasses). Store methanol in a flammable storage cabinet away from oxidizers and sources of ignition.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

Many parts of the GC and autosampler operate at temperatures high enough to cause serious burns. Allow heated zones to cool below 50°C before working on or around them.

Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must either be capped or have a column connected at all times.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Environmental Management System* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure because the impact of such a change on the chemistry of the procedure must be assessed prior to implementation.

Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever

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possible. However, do not change the concentrations of standards and reagents specifically designated in this SOP.

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedures*. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

This procedure produces the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-hazardous Waste	Not applicable
Aqueous waste (wastewater)	Hazardous Waste	Toxic
Methanol waste	Hazardous Waste	Flammable, toxic

5 SAMPLE HANDLING AND PRESERVATION

5.1 Containers and Required Sample Volume

5.1.1 Water Samples:

Water samples should be collected in pre-cleaned 40-mL screw cap vials equipped with Teflon-faced silicone septum. Volume collected should be sufficient to allow for replicate analysis yet minimize waste disposal. Three 40-mL vials should be sufficient to meet these objectives.

Water samples should be preserved with hydrochloric acid (HCl) to pH \leq 2 at the time of sampling. If an unpreserved water sample is received, inform the Chemistry Technical Director immediately so that the proper notifications can be made.

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5.1.2 Soil Samples:

Soil samples should be collected using 5 g EnCoreTM samplers when possible. Alternative collection methods are allowable if storage conditions and holding times conform to requirements. The following table summarizes the available techniques for soil samples:

Field Sampling, Storage, and Transport Options								
Collection	Field	Field/Transport	Holding Time	Laboratory				
Container	Preservation	Temperature	Under Field	Options				
			Storage Temp.	(see below)				
Encore*	None	$4 \pm 2^{\circ}$ C or < -	48 hours	1				
		7° C						
40-mL vial	None	$4 \pm 2^{\circ} \text{ C or } < -$	48 hours	1				
		7° C						
40-mL vial	Methanol	$4 \pm 2^{\circ}$ C or \leq -	14 days	2				
		7° C						
		Laboratory Optio	ns					
	If not ana	lyzed within 48 hours	of sampling.					
Option	Laboratory	Lab Storage	Holding	Time				
	Preservation	Temperature	(collection to analysis)					
1	Methanol	$<$ -7° C and \ge -	14 days					
		20 C						
2	As received	$<$ -7° C and \ge -	14 da	ys				
		20 C						

^{*} Samples received in Encore samplers are transferred to 40-mL vials prior to completing laboratory options or analysis within 48 hours of sampling.

Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, and minimize waste disposal. Three 5-g Encore containers or samples extruded into 40-mL VOA vials should be sufficient to meet these objectives.

Use of sediment jars, 25g EnCore samplers, or other methods of unconsolidated soil sample collection is unacceptable due to the substantial loss of volatile organics under these conditions. Request guidance from the supervisor or Technical Director if samples are received for analysis in these containers.

Note: A separate 10 gram aliquot of solid material should be collected for percent solids determination if results are required on a "dry weight" basis.

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5.1.3 Waste samples:

Waste samples (non-aqueous liquids, solid matrices other than soils) should be collected in a pre-cleaned 40-mL screw cap vial equipped with a Teflon-faced silicone septum vial. The vial should be filled to minimize headspace and stored or analyzed as outlined above. Only one vial need be collected as the volume will be sufficient for repeated analysis.

In general, storage, preservation, and holding times for wastes are the same as for soil samples (see section 5.1.2). However, if high concentrations of target analytes are suspected, waste samples should be segregated from low concentration volatile samples to prevent cross contamination, additionally, waste samples must be reviewed on a case-by-case basis and diluted to prevent damage to instrumentation due to either matrix interferences (e.g. foaming or volatile acids) or high concentrations of target analytes that could contaminate instruments. Non-aqueous liquids are frequently highly contaminated and should be preserve or dilute such that the sample to solvent ratio is 100 mg to 1 g in 10 mLs of methanol instead of the usual 5 grams. The original sample should be frozen.

5.2 Internal Chain-of-Custody

Verify sample IDs and dates and times of collection against the chain-of-custody form. Notify the sample custodian of any discrepancies.

Update the LIMS database internal custody form when sample containers are moved from the designated sample location. Change the container disposition to "active out" and the location to the appropriate room number. At the end of the day, return sample containers to the "Home" locations. Update the LIMS database using the "return to home location" feature and update container disposition to "available in". Verify that your initials are recorded whenever you update the LIMS custody information.

Samples are delivered by the Sample Custodian to Room 201, logged into the Laboratory Information Management System (LIMS), and transferred to the sample refrigerator (or freezer, as applicable). The LIMS database is then updated to change the container disposition to "available in".

Check the following information to ensure that the information on the sample containers corresponds to the information on the tracking sheets and the chain-of custody record:

- EPA Work Order Number
- EPA sample number.
- Case number.
- Sample Delivery Group number

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5.3 Preservation Verification

Verify that the samples are preserved as outlined in section 5.1.1 or 5.1.2.

5.4 Sample Storage

Water samples must be stored at >0 and <6 ° C. See section 5.1.2 for soil and waste storage requirements.

Maintain additional aliquots under these storage conditions until the holding time has expired <u>and</u> analysis is completed.

Retain samples and extracts for 60 days after the final analytical report is sent to the data user. This allows for confirmation of labeling information should questions arise. Storage conditions do not need to be monitored or comply with the above criteria.

5.5 Holding Time

Acid preserved water samples must be analyzed within 14 days from the date of sample collection; unpreserved water samples must be analyzed within 7 days from the date of sample collection. See section 5.1.2 for soil and waste holding time requirements.

6 INTERFERENCES

Method interference may be caused by impurities in the purge gas, organic compounds out gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory method and instrument blanks. The use of non-polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during storage and handling.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105° C. The trap and other parts of the system are also subjected to contamination; therefore, frequent bake out and purging of the entire system may be required.

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Solvents and other compounds, which are target analytes must never be introduced into the laboratory where volatiles analysis is performed. Dichloromethane, acetone and other common laboratory chemicals are target analytes under this SOP and must be excluded from Room 201. Particular attention must be paid to the possibility of transport of solvent vapors on individuals. Analysts should never enter Rooms 201 or 209 after being in the extraction laboratories or glassware washing area.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. All equipment, reagents, standards, and supplies must meet the technical and QC requirements of the reference method. Substitutions may be made provided that they are documented and equivalency is maintained.

7.1 Instruments and Equipment

- Analytical balance capable of measuring differences of 0.01 g.
- Gas chromatograph (Agilent AG6890 gas chromatograph, Agilent AG7890 gas chromatograph, or equivalent) equipped with a FID detector (or PID/FID in series) and a splitless injection port or an OI Low-Dead-Volume Injector, or equivalent.
- Data Acquisition and Processing System able to control the GC and to acquire, store, and process gas chromatographic data. The software must be able to calculate calibration factors and the concentrations of analytes in samples. Agilent Technologies EnviroQuant ChemStation software and data acquisition computers (or equivalent).
- Fused Silica Capillary Gas Chromatography Column 75m x 0.53mm x 3µm RTX-624 wide bore capillary column (Restek part # 10974, or equivalent). Any capillary column that provides adequate resolution, capacity, accuracy, and precision may be used.
- Purge and trap concentrator OI Eclipse 4660, Tekmar 3000 purge and trap concentrator, or equivalent.
- Autosampler: OI Archon 4552, Varian Archon, or equivalent.
- VOCARB 3000 type K trap, EST Vocarb 300 cat # E70300-K03, or equivalent alternate traps may be used provided that the adsorption and desorption characteristics obtained achieve equivalent or better method sensitivity and precision.

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7.2 Reagents

All chemicals must be entered into the EMS inventory upon receipt. The analyst must enter all reagent receipt and preparation in the LIMS.

- Methanol (Purge & Trap grade).
- Hydrochloric acid (HCl), concentrated, reagent grade
- Hydrochloric acid, 6N HCl Slowly add 250 mL of reagent grade concentrated HCl to 250 mL of organic-free method blank water. Prepare as needed, not to exceed annually. Also known as 1:1 or 1+1 HCl.
- Organic-free method blank water (prepared using USEPA Region 9 Laboratory SOP 205).

7.3 Standards

All standards must be entered into the EMS inventory upon receipt. The analyst must enter all standards into the LIMS. Standards and samples must not be stored in the same refrigerator or freezer. Protect all standards from light.

Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade materials. Store ampulated stock standard solutions in the Room 201 refrigerator at $>0^{\circ}$ to $<6^{\circ}$ C. Follow manufacturer's recommendations for solution expiration date.

Store all working standard solutions in glass bottles or vials with Teflon lined screw caps at <-10 $^{\circ}$ C.

Fresh standards should be prepared every six months, or sooner if comparison with check-standards indicates a problem. The standard solution must be checked frequently for stability. Replace all working standard solutions after six months or sooner if QC results indicate a problem.

Opened ampules of standards must be discarded six months after opening, at the manufacturer's expiration date, or earlier if evidence of degradation is observed. LIMS expiration dates must be revised when ampules are opened if the remaining time on the vendor expiration date exceeds six months. The vendor expiration date should be recorded in the comment portion of the standard record.

The following solution concentrations are recommended only; other concentrations and / or suppliers can be used.

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CAUTION: Allow all standard solutions to equilibrate to room temperature before use.

- Gasoline Stock Standard: Restek #30205 (XHc Unleaded Gasoline Composite Standard at 50,000 μg/mL), or equivalent. This standard is also used as the MS/MSD and LCS source for soil/solid matrices.
- <u>Gasoline Primary Dilution Standards (PDS)</u>: Prepare a solution by diluting the Gasoline Stock Standard to concentration 1,000 μg/mL in P&T methanol (200 uL to 10 mL), or equivalent.
- <u>SCV Stock Standard</u>: Supelco # 47516-U (Gasoline at 20,000 μg/mL), or equivalent.
- <u>SCV Primary Dilution Standards (SCVPDS)</u>: Prepare a solution by diluting the SCV Stock Standard to concentration 1000 μg/mL in P&T methanol (500 uL to 10 mL)), or equivalent.
- <u>Second Source Verification (SCV)</u> Equivalent to the mid-point initial calibration solution, 500 μg/L but prepared from a source different from the source of calibration standards (use SCV Primary Dilution Standards (SCVPDS) to make this standard).
- Window Defining Standard (WDS): AccuStandard Custom Mix S-10760-2.5X or equivalent. This solution contains 2-methylpentane and 1,2,4-trimethylbenzene at 5,000 µg/mL in methanol.
- <u>Window Defining Standard Primary Dilution Standards (WDSPDS)</u>: Prepare by diluting the WDS standard to 1000 μg/mL in P&T methanol (1000 uL to 5 mL), or equivalent.
- Surrogate Spike Stock: Restek # 30083, solution of α , α , α -trifluorotoluene in methanol at 10,000 µg/mL, or equivalent.
- Surrogate Spike: Prepare a solution by diluting the Surrogate Spike Solution Stock Standard to concentration of 625 μ g/mL in P&T methanol (625 uL to 10 mL), or equivalent.
- As needed, other hydrocarbon mixtures at 500 μ g/L in water. These standards are used for identification purpose only.
 - o Kerosene, 50 mg/mL; Restek 31256
 - o Diesel, 50 mg/mL; Restek 31258
 - o Stoddard, 20.0 mg/mL; AccuStandard HS-005S-40X

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- o Lacquer Thinner, 20.0 mg/mL; AccuStandard HS-001S-40X
- o Mineral Spirits, 20.0 mg/mL; AccuStandard HS-002S-40X
- o Naptha, 20.0 mg/mL; AccuStandard HS-003S-40X
- o Turpentine, 20.0 mg/mL; AccuStandard HS-004S-40X
- Hydraulic Fluid, 20.0 mg/mL; AccuStandard FU-020-D-40X
- o Turbine (Jet) fuel comp. 20.0 mg/mL; AccuStandard FU-006-40X
- Site specific hydrocarbon standards may also be supplied by the project manager for use in the laboratory.

7.3.1 Calibration Solutions

The following calibration solution concentrations are typical concentrations only; other concentrations may be used. Use of gastight syringes is required for sample and standard preparation.

Prepare all working calibration standards in organic-free method blank water.

Prepare the calibration standards by adding the following solutions to 50 mL of water in a gastight syringe to make standards at recommended concentrations prior to mixing and transferring the resulting solution to a 40-mL VOA vial and loading the vial onto the autosampler.

QC Type	Solution	Conc. µg/mL	Volume Used, µL	Final Volume, mL	Final Conc., µg/L
WDS	WDSPDS	1000	25	50	500
ICAL 1 QLS	Gas PDS	1000	2.5	50	50
ICAL 2	Gas PDS	1000	5	50	100
ICAL 3 CCV/LCS	Gas PDS	1000	25	50	500
ICAL 4	Gas PDS	1000	50	50	1,000
ICAL 5	Gas PDS	1000	100	50	2,000
SCV	SCV PDS	1000	25	50	500

7.4 The autosampler introduces 1 uL of the Surrogate Spike Solution to each sample at 625 μg/mL in P&T methanol for a final concentration of 125 μg/L.

7.5 Supplies

• Disposable Pasteur pipettes.

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- Fritted sparge vessels (10 mL and 25-mL).
- Gastight syringes (5-μL, 10-μL, 25-μL, 50-μL, 100-μL, 250-μL, 500-μL, 1-mL, 5-mL, 25-mL, and 50 mL). All syringes utilized must be rinsed with Purge and Trap grade methanol.
- pH paper (pH 0-14 range).
- Screw-cap vial, 40-mL VOC vials for standards with screw-hollow cap lined with 22 mm PTFE-faced silicone septa.
- Stainless steel spatulas
- Trap (VOCARB 3000 type K, or equivalent).
- Volumetric flasks, Class A appropriate sizes with ground glass stoppers.
- EnCore extrusion tool.
- 10 mL Disposable syringes.
- 40-mL VOC vials for standards with screw-hollow cap lined with 22 mm PTFE faced silicone septa.

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Note: If the instrument is equipped with a functioning PID, the detector should be calibrated for the surrogate. The information provided by the more selective detector can be used to verify that the presence of aromatic hydrocarbons even in the presence of significant amounts of other, i.e., straight chain hydrocarbons. However, the surrogate should always be reported from the FID; the PID calibration is not required for method control (i.e. do not stop analysis or rerun samples for PID failure).

Set up instruments using operating parameters provided in Appendix D. Adjust as needed to meet method and SOP requirements and chromatographic practice.

Enter data into ChemStation using file naming conventions provided in Appendix E.

Bake the trap and the GC oven for at least 14 minutes each day before samples are analyzed.

Prior to analyzing calibration, QC, or field samples, make a LIMS batch and sequence as required to obtain LIMS assigned IDs for the calibration and QC samples.

Ensure that all appropriate waste containers are properly connected and labeled.

8.2 Calibration and Standardization

The calibration standards preparation is detailed in Section 7.3.1. The same calibration is used for water and soil samples.

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Set up the purge and trap concentrator for water analysis ensuring that the sparge needles reach to within 5 mm of the bottom of the sparge cells. The same calibration is used for the analysis of both water and soil methanol extracts.

8.2.1 Initial Calibration

The retention time range for GRO is defined during initial calibration. Two specific gasoline components are used to establish the range, 2-methylpentane and 1,2,4-trimethylbenzene. The retention time range is then calculated based on the lower limit of the RT window for 2-methylpentane and the upper limit of the RT window for 1,2,4-trimethylbenzene.

Perform an initial calibration using standards prepared in Section 7.3.1. Refer to Section 9.2.1 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

Check that compound type in ChemStation is set to H. This setting sums the area between the start and end of the analyte range.

An example of initial calibration sequence appears below:

	Sample Name		Sample Name
1	IB	6	ICAL 4
2	WDS standard	7	ICAL 5
3	ICAL 1 (QLS)	8	IB
4	ICAL 2	9	SCV
5	ICAL 3 (CCV)	10	Other fuels as needed

Spike the water with the appropriate amount of primary dilution standard for the specific calibration solution being analyzed. See Section 7.3.1 for details.

Quantitate each calibration standard and samples using this retention time range established using WDS standard.

- 1. In the ChemStation data analysis module, load the current initial calibration method as outlined in the "ChemStation File Naming Convention"
- 2. Perform an initial calibration using the recommended concentrations listed in Section 7.3.
- 3. Update the response factors in the method using the newly acquired calibration files.
- 4. Update the retention time for the surrogate in the method using the newly acquired continuing calibration level.
- 5. Save the method as outlined in the "ChemStation File Naming Convention"

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- 6. Generate "Response Factor Report."
- 7. Check the calibration files listed on the "Response Factor Report" to insure that the correct files are being used.
- 8. Check the time and date to ensure that the correct update is used.
- 9. Process the SCV with the newly created initial calibration, check to make sure the "QLast Update" time and date match the "Response Factor Report".
- 10. Verify that the method was updated correctly.
- 11. Print page 3 of the ChemStation ICAL for gasoline to show that the method was updated correctly.
- 12. Copy the method to the LAN as outlined in the "ChemStation File Naming Convention"
- 13. Manually calculate a result for the surrogate in the SCV to insure that the correct RFs are being used and write the results on the quantitation report.
- 14. Save a hard copy of the initial calibration files so they may be copied and included in associated packages.

If the initial calibration, the SCV, and the IB meet all criteria specified in Appendix C, the remainder of the 12-hour analytical period may be used for the analysis of field and QC samples.

As needed, analyze other hydrocarbons with each ICAL.

8.2.2 Retention Time Windows

Based on experience and historical data, the EPA Region 9 Laboratory uses a default retention time window of +/- 0.03 minutes for single component analytes. This approach is based on the insignificant retention time drift observed historically and the option listed in SW846 Method 8000C section 11.6 to select an alternative approach to the usual analysis of standards for the calculation of retention time windows.

Establish the ChemStation retention time window as \pm 0.06 minutes for the surrogate (peaks that drift more than 0.03 minutes will be flagged "f" by the data system as possible false positive).

All surrogates in the field and QC samples must fall within the established retention time windows.

All target analytes, surrogates, and/or n-alkanes in the calibration verification analyses must fall within previously established retention time windows. If the retention time of any analyte does not fall within the established window, then corrective action must be taken to restore the system or a new calibration curve must be prepared for that compound.

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8.2.3 Continuing Calibration Verification (CCV)

Perform calibration using standard prepared in Section 7.3.1. Refer to Section 9.2.3 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

- 1. In the ChemStation data analysis module, load the current initial calibration method.
- 2. Acquire the continuing calibration using the current initial calibration method.
- 3. Quantitate the continuing calibration file.
- 4. Generate "Evaluate Continuing Calibration Report".
- 5. Manually calculate the result for the surrogate to insure that the correct RFs are being used. Record the result on the quantitation report.
- 6. As each run is quantitated during the day, make sure that the same date and time stamp, (e.g. "QLast Update: Mon Jul 25 08:15:58 2011"), is recorded on each file header.
- 7. If QLast Update time stamp changes, state the reason, repeat steps 4-6, and include the reports generated in the package.

Refer to Appendix C for frequency, acceptance criteria, and corrective action requirements.

8.2.4 Quantitation Limit Verification Standard (QLS)

Analyze a quantitation limit standard using standard prepared in Section 7.3.1. Refer to Section 9.2.4 and Appendix C for frequency, acceptance criteria, and corrective action requirements.

Refer to Appendix C for acceptance criteria and corrective action requirements.

8.3 Sample Analysis

Check that the LIMS ID and client sample ID on the vials coincide with the numbers on the LIMS Work Order to ensure that the correct sample is being analyzed.

If the sample has an unusual color, or other physical characteristic such as more than one phase, the presence of a precipitate, unusual viscosity, or physical signs of contamination. A screening analysis is required to protect the analytical system from damage or contamination and to determine the appropriate subsequent dilution. If an initial screening is necessary, analyze the sample at a 1:50 dilution, unless the group leader or Technical Director specifies otherwise. Document observed anomalies in the LIMS MMO field.

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MS/MSD and LCS spiking is preformed at time of analysis. CCV and LCS requirements maybe satisfied in one analysis.

8.3.1 Water Sample Preparation

Note in the LIMS MMO field in the work order window if there is headspace present in the sealed sample vial. If the bubble exceeds 6 mm in diameter, data qualification may be required.

Water Spike Amounts

Sample	Surrogate Spike, µL*	Gas PDS, μL
Blanks	1.0	NA
Samples	1.0	NA
CCV/LCS	1.0	25.0
MS/MSD	1.0	20.0

Prepare LCS in 50-mL gas-tight and transfer to 40-mL vial.

- Allow the samples to reach ambient room temperature before analysis.
- Break the chain of custody seal on the vial with a scalpel or other appropriate implement, and note if the seal is missing or compromised in any way.
- To spike the MS and MSD, quickly remove the cap from each 40-mL sample vial and spike directly in the field sample vial and recap the vial.
- Load the samples in the autosampler. The autosampler will add surrogate.
- After analysis, check the pH of the sample using pH 0-14 range pH paper. Record the pH in the injection logbook. Note any samples that have a pH greater than 2 in the LIMS MMO field in the work order window.

8.3.2 Soil Sample Preparation

The typical sample weight is 5 g (nominal).

The percent moisture is determined from a separate aliquot as described in EPA Region 9 Laboratory SOP 460, *Percent Moisture Determination*.

To prevent the loss of certain volatile organics the sample must not be allowed to reach room temperature. The following steps must be taken as rapidly as

NA = Not Applicable

^{*} SS is added by autosampler.

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possible to prevent loss of volatile components. Have the LIMS benchsheet prepared and all vials, standards, etc. ready prior to beginning the next steps.

- 1. Remove the sample from the refrigerator or freezer immediately prior to extraction or analysis. Samples should be extracted as soon as possible after receipt even if analysis will not to be performed immediately.
- 2. Break the chain of custody seal on the container with a scalpel or other appropriate implement, again making note in the logbook if the seal is missing or compromised in any way. Observe the sample closely for evidence of contamination. If the sample appears to contain hydrocarbons (an oily appearance or sheen), the sample must be analyzed at a dilution to prevent damage to the analytical system.
- 3. If possible, all samples within a sample delivery group should be extracted at the same time along with the MB preparation.
- 4. To extract samples collected with the EnCoreTM sampling device, transfer the contents of the EnCoreTM sampler into a 40-mL tared vial.
- 5. To extract bulk samples, use a stainless steel spatula. Immediately transfer approximately 5 g into a 40-mL tared vial.
- 6. Record the weight of soil added to the container to the nearest 0.01 g in the Volatile Extraction Logbook. Weight information is also entered in LIMS bench sheet at time of analysis.
- 7. Quickly add 10.0 mL of purge and trap grade methanol to the vial.
- 8. Prepare a soil MB adding approximatly 10 mL of purge and trap grade methanol used to extract field samples to the vial.
- 9. Store extracted samples in the laboratory freezer. Use this extract for the analysis and any subsequent dilutions that may be necessary.

8.3.3 Analytical Sequence and Sample Analysis

Set up a ChemStation data acquisition sequence from the LIMS sequence using the GC operating parameters in Appendix D. Include the client sample ID and the laboratory sample ID in the sample description field. Additional header information shall include the dilution factor, instrument ID, and the analyst's initials. Enter this sequence in the instrument run log, if used.

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For water analysis:

- 1. The method defaults are 5 mL sample, 5 mL final volume, and dilution factor 1. These values produce correct reporting limits but are only a starting point for data entry.
- 2. Batch samples as usual and leave the initial and final values in the batch as 5 mL and 5 mL respectively.
- 3. In the bench sheet, the sample initial and final volumes default to 5 mL.
- 4. Edit the bench sheet initial volume for samples and QC to reflect the actual volumes used.
- 5. When setting up the ChemStation sample sequence, enter 1 as the dilution factor in the multiplier field.

For soil analysis:

Spike MS/MSD samples with 5 uL of the Gasoline Stock Standard containing $50,000 \, \mu g/mL$ of gasoline to the vial containing the sample and methanol. Cap the vial and vortex for 30 seconds. These steps must be done rapidly in order to prevent the loss of volatile organics from the sample.

Prepare the soil method blank by adding 1 mL of the methanol used in preserving the soil samples in section 8.3.2 to 49 mL of reagent water in a syringe. Mix and transfer the resulting solution to a 40-mL VOA vial and load the vial onto the autosampler.

Add 1 mL of the extract to 49 mL of reagent water in a syringe. Mix and transfer the resulting solution to a 40-mL VOA vial and loading the vial onto the autosampler.

- 1. The LIMS batch defaults are 5 g sample, 5 mL final volume, and dilution factor 100. These values produce correct reporting limits but are only a starting point for data entry.
- 2. Batch samples as usual and leave the initial and final values in the batch as 5 g and 5 mL, respectively. (Note that the 5 mL volume represents the volume of the aqueous solution analyzed, not the methanol extract volume.)
- 3. In the bench sheet, the sample weight defaults to 5 g. Edit the initial weight to the actual weight used as needed.
- 4. Edit the bench sheet final volumes for samples and QC from 5 mL to the actual volume of methanol used to extract the samples (which is usually 10 mL).
- 5. When setting up the ChemStation sample sequence, enter 50 as the dilution factor in the multiplier field. (Do not apply the multiplier field to sample or surrogate results in ChemStation; LIMS does this later). The dilution factor, D = syringe volume/methanol extract volume in the syringe. This is typically 50 mL/1 mL = 50. If a greater dilution is required, calculate the

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dilution factor based on the volume of methanol used, e.g. for 100 uL, D is 50 mL/0.1 mL = 500.

See Section 9.3 for batch quality control (QC) frequency and corrective action requirements. It is highly recommended that the MB, LCS, and MS/MSD be analyzed as early as possible in the analysis of a batch to allow for time to check QCs.

Example Field Sample Analysis Sequence:

	Sample Name		Sample Name
1	Prep (as needed)	6	MSD (as needed)
2	CCV/LCS*	7-18	Field samples (as needed)
3	QLS	19	IB (as needed)
4	MB/IB	20	CCV
5	MS (as needed)		

^{*} CCV and LCS requirements may be satisfied in one analysis.

Enter the first and last sample positions in the concentrator and, with the ChemStation software in data acquisition mode, press the start button on the concentrator to begin purging the first sample. The purge and trap concentrator parameters are found in Appendix D.

When possible or if there are indications that the sample may foam, observe the initial purging. If the sample does foam, it can be analyzed as long as the foam does not enter the sparge vessel neck and enter the transfer line leading to the trap.

If it appears that the sample will foam excessively, discontinue the purging by pressing the [2nd], [on], and [enter] keys. Drain the sparge cell; rinse it with methanol, then reagent water. Place the sample waste and rinsate in the aqueous waste container. Bake out the trap and the GC for 25 minutes before analyzing additional samples. Analyze reagent water blank to show that the sampler is free from contamination before analyzing sample.

Analyze the sample at a 1:10 dilution, or other appropriate dilution to prevent foaming even though the detection limits are elevated. Document any sample foaming in the run log and the LIMS MMO field.

8.3.4 Analyte Identification and Quantitation

Update the center of the retention time window for the surrogate by using the absolute retention times from the calibration verification standard at the beginning of the analytical shift.

^{**} MB and IB requirements may be satisfied in one analysis.

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All surrogates in the field and QC samples must fall within the \pm 0.03 minute retention time window or the analyst must reject the analysis or review and accept the data with a written comment such as "retention time shift due to high concentration of interfering hydrocarbon".

If the retention time does not fall within the retention time window and no source of drift is identified, then take corrective action to restore the system. If repairs to the system are required then a new initial calibration must be performed.

Review the sample chromatograms for appropriate qualification. Several situations are routinely encountered:

- The chromatographic pattern resembles the standards; proceed with quantitation and reporting.
- The chromatogram differs markedly from the standard; visually compare the sample chromatogram to available hydrocarbons analyzed with the initial calibration. Software tools such as overlaying various standard chromatograms on the sample chromatogram should be employed when helpful or to support the qualification.
- All detected results should be flagged in LIMS to qualify the reported value. When possible, indicate the product or fuel type.
- The majority of the area is due to a single-component and not a hydrocarbon mixture; consult the group leader and/or technical director to determine if the peak should be excluded from the quantitation. Use the WO Memo field and appropriate qualifier flags to report the data.
- The client has requested qualitative review against various standards; refer to the TDF and project notes to determine the project specific analysis and reporting procedures. This may include calibrating with a source supplied by the client, review against numerous standard chromatograms, or other procedures. Always document this process in the WO Memo field and in the data package and report as necessary

Quantitate the sample data using the ChemStation software using the appropriate initial calibration mean CFs. If applicable, indicate degree of similarity of sample chromatogram to the gasoline standard. Print out quantitation reports and chromatograms for each field and QC sample.

LIMS calculates final analyte concentrations in samples. To verify the LIMS reported values for water samples, calculate results for target analytes using the following equation:

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8.3.4.1 Water Calculations

Calculate target analyte concentrations in aqueous samples using Equation 1.

Equation 1:

Concentration (ug / L) =
$$\frac{A_x \times DF}{RF}$$

Where:

 A_x = area response for analyte x

DF = dilution factor

RF = mean response factor from the initial calibration (area/concentration)

8.3.4.2 Soil Calculations

Calculate target analyte concentrations in soil samples using Equation 2.

Equation 2:

Concentration (mg/Kg dry weight basis) =
$$\frac{A_x \times V_t \times DF \times V_p \times 1,000}{RF \times W \times D \times V_i \times 1,000}$$

Where:

 A_x = area response for analyte x

D = dry weight factor (Percent solids/100)

W = weight of sample in grams

RF = mean response factor from the initial calibration (area/concentration)

 V_t = total volume of extract in mL (see note)

DF = dilution factor (volume of water divided by volume of

extract (nominally 5 mL / 0.1 mL or 50)

 V_i = volume of extract injected in μL

Vp = volume of extract purged in mL (i.e. 5 mL)

1,000 (in numerator) $1000 \mu L = 1 mL$

1,000 (in denominator) 1000 mL = 1 L

Yields concentration units of $\mu g/g = mg/Kg$

Note: V_t is equal to the volume of methanol (see benchsheet) plus the volume of water from the solid calculated as (1 - % solid) * W. See EPA Region 9 Lab SOP 460 for percent solids determination.

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8.3.5 Manual Integration

Review the baseline drawn by the data system integrator to verify that it accurately reflects the area response of the sample components. If in the judgment of the analyst, it does not, then correct the integration using the ChemStation QEDIT software module. Document manual integrations, if any, following the procedure described in USEPA Region 9 Laboratory SOP 835, *Chromatographic Integration Procedures*.

8.3.6 Data and QC Review

As soon as possible after analysis (typically prior to entry into LIMS), inspect sample and QC data for compliance with QC limits in Appendix C. If no significant problems are found, review the following QC data for compliance with SOP requirements:

- Target analyte results must be within range of initial calibration.
- Process and review the results for the IB, CCV, and QLS instrument QC samples. Print a ChemStation Evaluate Continuing Calibration Report using the appropriate settings to verify that the CCV and QLS QC sample results are within QC limits. See Section 9.3 for instrument QC requirements.
- Process and review the results for the MB, LCS, and MS/MSD batch QC samples and verify that the results are within QC limits. See Section 9.3 for batch QC requirements.
- Check that surrogate compound retention times are within the window specified in Section 9.4.1 and Appendix C. Determine if surrogate recoveries for field and QC samples are within QC limits. See Section 9.4 for sample QC requirements.
- Review all sample results to determine if any samples need to be reanalyzed at a dilution. If any of the target compounds in soil extract or water samples exceed the initial calibration range of the instrument, dilute by using a smaller aliquot of the water sample or soil extract combined with reagent water.
- If a run is rejected for any reason, mark the raw data "Not Used" in large print and document the reason on the quantitation report. Initial and date the comment.

8.3.7 Data Export and LIMS Entry

Export data from the instrument into text files. Import into the LIMS using

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DataTool. .Review final results in the LIMS. The LIMS will report two significant figures and detected results to one-half the QL. The LIMS will flag values between one-half the QL and the QL as estimated (J). The analyst must manually add a qualifier flag (C1) indicating that the reported concentration is estimated because it is less than the quantitation limit. Qualify data based on QC results and guidelines in the EPA Region 9 Laboratory QA Plan.

- 1. Generate epatemp.txt files for field and QC samples by also printing the report to the screen; these files are used by the LIMS DataTool module to import the instrument results into the Data Entry/Review table.
- 2. Copy data files from the local drive to the appropriate instrument data subdirectory on the Region 9 LAN to make them available to LIMS and for archiving.
- 3. Create an empty upload file containing the samples analyzed in the LIMS batch or sequence. Import and merge the data files using the LIMS DataTool module. Load the resulting merged data file into the LIMS Data Entry/Review table.
- 4. Populate the empty LIMS sequence with the samples actually analyzed by editing the empty LIMS sequence; import the sample information using Data Tool.
- 5. After making an empty upload file containing the samples analyzed in the LIMS batch or sequence, import and merge the data files using the LIMS Data Tool module. Load the resulting merged data file into the LIMS Data Entry/Review table.
- 6. In order to take the dilution that occurs during soil sample preparation into account, the dilution factor for undiluted soil samples in the LIMS Data Entry/Review table must be 50. Any actual sample dilutions must be multiplied by 50 to obtain the effective sample dilution to be entered in LIMS. Edit dilutions in DataTool or LIMS entry table as needed.
- 7. Review results in the LIMS. The LIMS will report two significant figures and detected results to one-half the QL. The LIMS will flag values between one-half the QL and the QL values over the calibration range as estimated (J). The analyst must manually add a qualifier flag (C1 or C2) indicating that the reported concentration is estimated because it is less than the quantitation limit or exceeds the calibration range. Qualify and flag results in the LIMS Data Entry/Review table following Appendix M of the Region 9 Quality Assurance Manual.

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8.4 Maintenance

The analyst should observe trends in the data such as declining response, erratic relative response, loss of classes of compounds, etc., which may signal the need for instrument maintenance. Document all routine maintenance or corrective actions taken in the maintenance logbook. Preventative maintenance procedures are listed in Appendix F.

The following sections describe possible causes and corrective actions for common problems. Refer to Appendix F for routine preventative maintenance procedures and schedule.

8.4.1 Purge and trap maintenance

Symptom:

Carryover

Possible causes: Cold spot in system, especially the transfer lines between the sparge unit and the concentrator or between the concentrator and the GC or analyzing a sample containing high molecular weight components or analyzing high-level and low-level samples sequentially. Corrective action: Check temperatures of all heated zones. Adjust temperatures or replace heaters as required. Flush valve, gas lines, and sample lines with methanol or reagent water and bake out.

• Loss of sensitivity to selected analytes and increased pressure to maintain purge flow.

Possible cause: Degradation of trap. Corrective action: Replace trap.

• Loss of all purged analytes.

Possible cause: Leak in system.

Corrective action: Leak check purge and trap system. Inspect sparge ferrules and replace them when worn or distorted.

8.4.2 GC Maintenance

Symptom:

Carryover

Possible causes: Analyzing a sample containing high molecular weight components or analyzing high-level and low-level samples sequentially. Corrective action: As necessary, replace inlet liner, clean inlet, bake out inlet, bake out column, clip column, replace septum, replace column.

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• Shorter retention time.

Possible cause: column flow rate problem.

Corrective action: check flow rate and adjust as necessary.

• Longer retention time and or smaller peaks.

Possible causes: column flow rate problem, injection port leak, or column contamination.

Corrective action: as necessary, check for leaks, replace septum, replace the liner, replace the lower injection port seal, and cut the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

Loss of resolution.

Possible causes: column flow rate problem, injection port leak, or column contamination.

Corrective action: check for leaks, replace septum, replace the liner, replace inlet seal, and clip the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

9 QUALITY CONTROL

The EPA Region 9 Laboratory operates a formal quality control program and tracks compliance using the Lab QC Database. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. A summary of QC criteria is provided in Appendix C.

9.1 Demonstration of Capability (DOC)

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in EPA Region 9 Laboratory SOP 880.

9.2 Instrument QC

Acceptance criteria for QC parameters are listed in Appendix C of this document.

9.2.1 Initial Calibration

Demonstration and documentation of an acceptable initial calibration are required before any samples are analyzed

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The GC system must be calibrated whenever corrective action that changes instrument response (e.g., detector gas adjustment, column replacement, etc.) is performed or if the calibration verification criteria cannot be met.

• The data system calculates the calibration factor (CF) using Equation 3.

Equation 3

$$CF = (A_x)/(C_x)$$

Where

Ax = Area of analyte x, or area sum response of gasoline $Cx = Concentration of the standard injected (<math>\mu g/L$)

• The data system calculates the percent relative standard deviation (%RSD) of the CF values for each analyte using Equation 4.

Equation 4

$$%RSD = (SD/CF_{avg}) \times 100$$

Where SD is the sample standard deviation and is calculated as:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - CF_{avg})^2}{n-1}}$$

Where:

- Print a ChemStation Response Factor Report. Verify that the %RSD of the target analytes and the surrogate are within QC limits immediately after the initial calibration is finished. See Appendix C for QC limits.
- If an ICAL fails because of one standard, a fresh solution of that standard may be re-analyzed and substituted for the failed one in the ICAL. If more than one standard fails, corrective action is required.
- Analyze an SCV sample immediately after each initial calibration. Calculate the calibration factor (CF) for the target analytes and the surrogate compound using Equation 3.

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• Calculate the percent difference (%D) between the SCV CF and the initial calibration average CF for the target analytes and the surrogate using Equation 5.

Equation 5:

$$^{\circ}$$
_O $D = \frac{CF_c - CF_{avg}}{CF_{avg}} x 100$

Where:

$$CF_c = SCV \text{ or } CCV CF$$

 $CF_{avg} = ICAL \text{ mean } CF$

 See Appendix C for QC limits. If the SCV sample fails it may be repeated once. If the second SCV fails, the cause for failure must be determined and corrected before analysis of samples can proceed.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

9.2.2 SCV Analysis

Analyze an SCV sample immediately after each initial calibration. See Appendix C for QC limits. If the SCV sample fails it may be repeated once. If the second SCV fails, the cause for failure must be determined and corrected before analysis of samples can proceed.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

Note: The SCV may not serve as the blank spike (BS/LCS).

9.2.3 Continuing Calibration Verification

- Analyze a CCV standard at the beginning of each analytical period and at the end of the analytical period. The analytical period begins with the injection of the CCV standard and ends with the injection of the last sample that can be injected within the period.
- Calculate the calibration factor (CF) for the target analytes and the surrogate compound using Equation 3.
- Calculate the percent difference (%D) between the calibration verification CF and the initial calibration average CF for the target analytes and the surrogate using Equation 5.

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• The %D must be within QC limits. See Appendix C for QC limits. If an analyte fails this criterion, a second calibration verification may be analyzed. Repeated failure requires that corrective action be taken to restore the system before any additional samples are analyzed. All affected samples must be re-analyzed.

If repairs to the system are required, then a new initial calibration must be performed. The analyst should observe trends in the data such as declining response, erratic response, etc., which may signal the need for instrument maintenance.

• Acceptable sample analyses must be bracketed by the analyses of calibration verification standards that meet QC limits.

9.2.4 Quantitation Limit Standard

- Analyze a quantitation limit standard (QLS) each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit.
- Calculate the concentration of the target analytes using Equation 1.
- Calculate the percent of true value (TV) for the target analytes using Equation 6.

Equation 6:

% True Value =
$$(Cd / Tv) \times 100$$

Where:

Cd = Concentration determined by analysis

Tv = True value of standard

• If the % TV is not within the QC limits in Appendix C, analyze a second QLS sample. Repeated failure requires that the cause be determined and corrected before analysis of samples can begin. If repairs to the system are required, then a new initial calibration must be performed.

9.2.5 Instrument Blank

At a minimum, one acceptable IB is required for each analysis period.

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- Evaluate the IB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- If the IB results are not within QC limits, analyze a second IB. If the second IB also fails but the system is significantly cleaner, another IB may be analyzed; if not, take corrective action.
- Corrective action If the IB is not acceptable, the source of the contamination must be found and eliminated and the problem documented before analysis can proceed.
- Surrogate recovery is not evaluated for IB QC samples.
- The IB may serve as the Method Blank for water analysis or Method Blank for soil or water analysis if it contains the methanol used for extracting soil samples.

9.3 Batch QC

9.3.1 Method Blank

- Extract and analyze a method blank (MB) to demonstrate that the entire analytical system from extraction through GC analysis is free of contamination.
- For aqueous samples, a MB is identical to an IB. For soil sample analysis, it is necessary to prepare an extracted MB.
- Refer to Appendix C for frequency, acceptance criteria, and corrective action requirements.
- If the surrogate recovery does not meet acceptance criteria, re-analyze the MB. If the surrogate recovery still does not meet acceptance criteria, the cause should be determined and corrected before analysis of samples can begin.
- Evaluate the MB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- Corrective action if the MB result exceeds QC limits and the sample result is less than five times the MB analyte result, re-analyze the MB. If the MB result still exceeds QC limits, then the MB and all associated samples must be re-prepared and re-analyzed. If the MB result exceeds QC limits and the

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sample result is \geq five times the MB result or is not detected, then report the sample result.

9.3.2 Laboratory Control Sample

- Analyze a laboratory control sample (LCS) to demonstrate that the analytical system is in control. Since the CCV and LCS are prepared from the same source and level, the Continuing Calibration Verification run also serves as the LCS for aqueous samples, but the LCS uses historical control limits.
- Calculate the percent recovery (%R) using Equation 7.

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Equation 7:
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\% Rec = (LCS/SA)×100
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Where, LCS = LCS result SA = Spike added

- The %R must be within the QC limits in Appendix C.
- If the LCS recovery does not meet criteria provided in Appendix C, rerun the LCS once to verify. If still unacceptable determine the cause, take corrective action and document these actions.

9.3.3 Matrix Spike/Matrix Spike Duplicate

- Matrix spike (MS) and matrix spike duplicate (MSD) samples are extracted and analyzed for each batch extracted as a group. Matrix QC samples are usually designated in the field. In the event that a sample was not designated as the matrix spike sample and adequate sample volume exists, the analyst will choose one <u>representative</u> sample from the SDG for QC analysis. Do not choose any obvious field blanks or trip blanks as the QC sample.
- Calculate the recovery of each analyte using Equation 8.

Equation 8:

$$\% Rec = ((SSR - SR)/SA) \times 100$$

Where

SSR = Spiked sample result

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SR = Unspiked sample result SA = Spike added

• Calculate the relative percent differences (RPD) of the recoveries of each analyte in the MS and MSD using Equation 9.

Equation 9:

$$RPD = \frac{(MSC - MSDC)}{(MSC + MSDC)/2} \times 100$$

Where,

MSC = Measured concentration of analyte in MS MSDC = Measured concentration of analyte in MSD

• See Appendix C for QC limits.

The MS/MSD recovery limits are advisory limits only. If the limits are not met, then no further action is required, as long as the LCS is within limits, since the purpose of these analyses is to determine matrix effects on compound recovery. However, frequent failure to meet the recovery or RPD criteria should alert the analyst that a problem may exist and must be investigated. The analyst should analyze the matrix spike solution and check the recoveries of the spike compounds. A new solution should be prepared if the recoveries are not within 20% of expected.

• The table below lists the action to be taken based on the LCS and MS/MSD results.

QC ACCEPTANCE	MAT	RIX	+ = F	PASS		_=	FAI	L
CASE	1	2	3	4	5	6	7	8
LCS - % REC	+	+	+	+				
MS/MSD -% REC	+		+		+		+	
MS/MSD – RPD	+	+			+	+		

Case 1: Extraction batch acceptable.

Case 2: Extraction batch acceptable; matrix effect confirmed.

Cases 3 & 4: Extraction batch is unsatisfactory. Investigate MS/MSD problem and document findings in the LIMS memo field.

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Cases 5, 6, 7, & 8: Extraction batch rejected. If additional sample volume is available, the batch should be re-extracted.

9.3.4 Storage Blank

- Every Monday morning, or the first workday of the week, fill three 40-mL screw-cap volatile vials with PTFE-faced silicone septum with reagent water, acidify to $pH \le 2$, and store them with the samples in the sample storage refrigerator.
- Analyze storage blank (SB) once every week while samples are being stored waiting for analysis. The storage blank indicates whether contamination may have occurred during sample storage.
- If samples have been stored in the refrigerator during the previous week, analyze the storage blank the following Monday, or on the first work day of that week. If samples have not been stored in the refrigerator during the previous week, discard the blanks and place new storage blanks in the refrigerator.
- Evaluate the SB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.
- If the SB does not meet QC criteria, all affected data must be qualified.

9.4 Sample QC

9.4.1 Surrogate Recovery

• Calculate the surrogate recovery in all field and QC samples immediately after analysis, using the following formula:

Equation 10:

 $%R = (Amount Found/Amount Spiked) \times 100.$

- The surrogate recovery must be within QC limits. See Appendix C for QC limits.
- Take the following steps if surrogate recovery is not within the limits:

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- 1. If the system is equipped with a functioning PID, the second detector can be used to provide further information. If sample matrix is the source of the error, as demonstrated by acceptable surrogate recovery on the PID, document the issue in the WO MMO field and on the chromatogram and continue. If not, proceed with troubleshooting.
- 2. Ensure that there are no calculation errors and check the system performance.
- 3. Re-analyze the extract if a system performance problem or calculation error is not evident. The extract may be diluted for re-analysis if examination of the chromatogram so indicates.
- 4. If re-analysis of the extract does not solve the problem, the sample may have to be re-extracted. Corrective action is decided by the EPA Chemistry Technical Director on a case-by-case basis.
- Do not re-extract undiluted samples with surrogate recoveries outside the limits if the diluted analysis with acceptable surrogate recoveries is being submitted. Report the event in the run log.
- Do not re-analyze the MS/MSD samples, even if surrogate recoveries are outside the limits.
- If the sample associated with the MS/MSD analyses does not meet the surrogate recovery criteria, it should be re-analyzed only if the matrix spike and duplicate surrogate recoveries are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the matrix interference is confirmed and the sample does not need re-analysis.
- If the surrogate recoveries of the re-analysis of the extract are within limits, then:
 - 1. If the re-analysis was undiluted, the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. Mark the first chromatogram as "Not Reported, see reanalysis."
 - 2. If the re-analysis was diluted, the problem was a matrix effect. Report the results from the re-analysis and submit the data from both analyses and discuss the result in the LIMS WO MMO filed. Mark the first chromatogram as "Not Reported, see re-analysis."
 - 3. If the surrogate recoveries of the re-extraction are within limits, then the problem was within the laboratory's control. Report the results from the re-extraction, mark the first chromatogram as "Not Reported, see reanalysis."

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4. If the re-extraction does not solve the problem, report the results from the first analysis and submit the data from both analyses.

9.5 Method Performance

The following table summarizes method performance by matrix for the period 11/12/2012 to 11/13/2013.

Method Performance

Analyte	Matrix	QC	Number of	Number of Mean		95% Confidence
		Type	Measurements	Recovery,%	(σ)	Interval (2σ)
TPH-GRO	Water	LCS	27	99.9	5.74	88.4 - 111
TPH-GRO	Solid	LCS	32	98.3	6.76	84.8 - 112

The following functional areas of the SOP may be significant sources of analytical error:

- Poor purge efficiency due to specific analyte characteristics or other problems.
- Standard degradation.
- Volatile compound losses in spike solutions and standards.

10 DOCUMENTATION

10.1 Standards

All standards (ICAL, ICV/CCV, QL, MS/MSD, and LCS) are recorded in the Element database. A copy of each Analytical Standard Record associated with sample analysis must be included in the data package.

10.2 Reagents

Record all reagents used for each analytical batch in the LIMS.

10.3 Analytical sequence

The analytical sequence is documented in the LIMS or in the instrument Run Log. Case Number, SDG number, date of analysis, QC solution IDs, analyst initials, lab sample IDs, client sample IDs, dilution factors and comments, if any, are recorded.

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10.4 Analytical Report and Data Package

Analytical reports are produced using the LIMS. The data package is produced from LIMS and manual log records. EPA Region 9 Laboratory SOP 845 *Analytical Data Review* provides the typical format for data package deliverables.

10.5 Maintenance Logbook

Maintain a maintenance logbook for each instrument covered in this SOP. Document the following:

- Initial installation and performance.
- Subsequent instrument modifications and upgrades, including major software upgrades.
- All preventative or routine maintenance performed including repairs and corrective or remedial actions. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control.

All entries should be made in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control.*

10.6 SOP Distribution and Acknowledgement

After approval, distribute an electronic copy of the final SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. (The Lab QC Database contains a list of assigned analysts for each SOP). All approved EPA Region 9 Laboratory SOPs are maintained in the LotusNotes database in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

10.7 SOP Revisions

Revisions to this SOP are summarized in Appendix G.

11 REFERENCES

Agilent Technologies EnviroQuant ChemStation User's Guide

Agilent AG6890 Gas Chromatograph Users Manual.

Agilent AG7890 Gas Chromatograph Users Manual.

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Varian Archon Autosampler and HP 3000 Concentrator Operator's Manuals.

- OI 4660 and 4552 Operator's Manuals.
- U.S. Environmental Protection Agency, *Method 5030C*, *Purge-and-Trap for Aqueous Samples*, Revision 3, May 2003.
- U.S. Environmental Protection Agency, *Method 5035A*, *Closed-system Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Draft Revision 1, July 2002.
- U.S. Environmental Protection Agency, *Method 8000C, Determinative Chromatographic Separations*, Revision 3, March 2003.
- U.S. Environmental Protection Agency, *Method 8015C*, *Nonhalogenated Organics Using GC/FID*, Revision 3, February 2007.
- U.S. EPA Region 9 Laboratory SOP 205, *Preparation of Organic Free Method Blank Water*.
- U.S. Environmental Protection Agency Region 9 SOP 460, Percent Solids Determination

APPENDIX A. DEVIATIONS FROM THE REFERENCE METHOD

- 1. The CF is area/concentration unit (μg/L) not area/mass (ng) as in the reference method. The formulas for determining sample analyte concentrations have been modified to reflect this change.\
- 2. This SOP, following the EPA Region 9 Laboratory Quality Assurance Plan, specifies preservation and storage temperatures for water samples of $>0^{\circ}$ to $<6^{\circ}$ C, while the reference method specifies $<4^{\circ}$ C.

APPENDIX B. ANALYTES AND QUANTITATION LIMITS

Analyte	QL, 5g Solid,	QL, 5 mL
	mg/kg	Water, μg/L
TPH-GRO	5.0	50

APPENDIX C. QUALITY CONTROL MEASURES AND CRITERIA

QC Measurement	Frequency	Criteria
Initial Calibration (ICAL) RSD	Instrument setup, after maintenance, and when CCV fails criteria	≤ 20
Second Source Verification (SCV) %D	After each ICAL	± 30
Calibration Verification (CCV) %D	Beginning of each daily sequence, every 12 hours, end of sequence	±20
Quantitation Limit Standard (QLS)	After first CCV of sequence, and after 40 samples.	\pm 40% of TV
Blanks : MB IB SB	One per Batch After each CCV Once per week	< ½ QL
Laboratory Control Sample (LCS) %R	One per Batch	Water: 81 - 119 Soil: 78 - 119
MS/MSD %R	One per SDG	Water:66 - 148 Soil: 73-127
MS/MSD RPD	One per SDG	Water:5 Soil: 10
Surrogate Recovery of QC and field samples (except IB) %R	Every sample	Water:87 - 110 Soil: 76 - 124
Retention Time Windows	Each CCV	±.03 minutes

^{*}ChemStation window is ± 6 SD of RT Study (SD is usually 0.01 min) and peaks are flagged at the more restrictive window.

APPENDIX D. RECOMMENDED INSTRUMENT PARAMETERS

AG 6890 OR AG7890 Gas Chromatograph

INSTRUMENT CONTROL PARAMETERS

6890 GC METHOD

OVEN

Initial temp: 35 'C (On) Maximum temp: 260 'C Initial time: 3.00 min Equilibration time: 0.50 min

Ramps:

Rate Final temp Final time CRYO (N2)

1 10.00 240 2.00 Cryo: Off 2 0.0(Off) Cryo fault: Off

Post temp: 0 'C Cryo timeout: 120.00 min (Off)

Post time: 0.00 min Quick cryo cool: Off Run time: 25.50 min Ambient temp: 25 'C

FRONT INLET (SPLIT/SPLITLESS) BACK INLET (UNKNOWN)

Mode: Split

Initial temp: 225 'C (On) Pressure: 13.73 psi (On)

Split ratio: 2:1

Split flow: 20.0 mL/min Total flow: 32.9 mL/min

Gas saver: Off
Gas type: Helium

COLUMN 1 COLUMN 2

Capillary Column (not installed) Model Number: restek Rtx-624

75M, .53id, 3.0um Max temperature: 240 'C Nominal length: 75.0 m Nominal diameter: 530.00 um Nominal film thickness: 3.00 um

Mode: constant flow Initial flow: 10.0 mL/min Nominal init pressure: 13.73 psi Average velocity: 53 cm/sec

Inlet: Front Inlet
Outlet: Front Detector

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Outlet pressure: ambient

FRONT DETECTOR () BACK DETECTOR ()

Temperature: 280 'C (On) flow: 30.0 mL/min (On) flow: 450.0 mL/min (On)

Mode: Constant column+makeup flow

Combined flow: 30.0 mL/min

Makeup flow: On

Makeup Gas Type: Nitrogen

Flame: On Electrometer: On Lit offset: 2.0

SIGNAL 1 SIGNAL 2

Data rate: 10 Hz
Type: front detector
Save Data: On
Start Save Time: 0.70 min Zero: 0.0 (Off)
Stop Save Time: 22.50 min
Start Save Time: 0.70 min Zero: 0.0 (Off)
Stop Save Time: 22.50 min
Save Time: 0.70 min Zero: 0.0 (Off)
Stop Save Time: 22.50 min
Save Time: 0.70 min
Save Time: 0.70

Range: 0 Attenuation: 0

Fast Peaks: Off Attenuation: 0

COLUMN COMP 1 COLUMN COMP 2

Derive from front detector Derive from front detector

POST RUN

Post Time: 0.00 min

TIME TABLE

Time Specifier Parameter & Setpoint

7673 Injector

Front Injector:
Sample Washes 0
Sample Pumps 6
Injection Volume 1.0 microliters
Syringe Size 10.0 microliters
Nanoliter Adapter Off
PostInj Solvent A Washes 0
PostInj Solvent B Washes 0
Viscosity Delay 0 seconds
Plunger Speed Fast

Back Injector: Sample Washes 0 Sample Pumps 6 Injection Volume 1.0 microliters Syringe Size 10.0 microliters Nanoliter Adapter Off PostInj Solvent A Washes 0 PostInj Solvent B Washes 0 Viscosity Delay 0 seconds Plunger Speed Fast

Column 1 Inventory Number: Rtx-624

Column 2 Inventory Number:

END OF INSTRUMENT CONTROL PARAMETERS

OI 4660 Concentrator

Recommended operating settings for the OI 4660 purge & trap concentrator that is interfaced with the HP 5890 Series II GC and the DPM-16 autosampler is as follows.

<u>PARAMETER</u>	<u>SETTING</u>
Purge temperature	20° C
Sample temperature	ambient
Purge Time	11 minutes
Dry purge	2.3 minutes
Purge Flow	35 - 40 mL/min
Desorb	2.00 minutes @ 240° C
Bake	14 minutes @ 240° C
Valve temperature	100° C
Mount temperature	40° C
Line temperature	100° C
DPM16 transfer line	100° C
DPM Valve temperature	100° C
Water Management	ON
Purge Temperature	100° C
Desorb Temperature	20° C
Bake Temperature	240° C

Recommended Purge and Trap Concentrator operating parameters

Tekmar 3100 Concentrator

The recommended operating method for the Tekmar 3100 A purge & trap concentrator which is interfaced with the AG6890 GC and Varian Archon Autosampler is as follows.

PARAMETER	SETTING
Standby temperature	32° C
Preheat temperature	N/A
Prepurge time	N/A
Sample temperature	Ambient for medium-level/20° C for low-level
Purge Time	11 min
Dry purge	0.6 min
Purge Flow	35 - 40 mL/min
Desorb preheat temperature	245° C
Desorb	2 min @ 250 ° C
Bake	10 min @ 260 ° C
Auto drain	On
Bake gas bypass	Off
Valve temperature	140 ° C
Mount temperature	100 ° C
MSS line temperature	40 ° C
Line temperature	140 ° C
Bottom of trap temperature	NA

Varian Archon/OI 4662 Autosampler

The recommended operating method for the Archon autosampler which is interfaced with the Tekmar 3000 and the HP5973 GC/MS is as follows.

PARAMETER		SETTING
Valve temperature	100 ° C	
Line temperature	110 ° C	

APPENDIX E. CHEMSTATION FILE NAMING CONVENTIONS

ChemStation File Naming Convention

Files for data, methods, tunes, and sequences on ChemStation computers and the LAN are named using the following naming conventions:

Directories

On the Workstation (When available, use D: drive):

Data: C:\MSDCHEM\1\DATA\YEAR\DATA\MMDDYYSS or

D:\MSDCHEM\YEAR\DATA\MMDDYYSS

Methods: C:\MSDCHEM\1\DATA\YEAR\METHODS or

D:\MSDCHEM\YEAR\METHODS

Sequences: C:\MSDCHEM\1\DATA\YEAR\SEQUENCE or

D:\MSDCHEM\YEAR\SEQUENCE

Tunes: C:\MSDCHEM\1\5973N or C:\MSDCHEM\1\5975

On the LAN:

Data: I:\DATA\ROOM NUMBER\INSTRUMENT\YEAR\DATA\MMDDYYSS

Methods: I:\DATA\ROOM NUMBER\INSTRUMENT\YEAR\METHODS Sequences: I:\DATA\ROOM NUMBER\INSTRUMENT\YEAR\SEQUENCE

Tunes: I:\ DATA\ROOM NUMBER\INSTRUMENT\YEAR\TUNE

Methods

MMDDYYATC

Sequence

MMDDYYCSS

Data Files

MMDDYYCSS

Tune Files

MMDDYYA

Variables

A: Enter analysis, as follow:

504 EDBTO15 TO15BNA BNA

BNA (SIM) PAH or PCP

PEST PEST
PCB PCB
RSK175 RSK
TPH-G GRO
TPH-D DRO

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VOA VOA BFB BFB DFTPP DFT

C: Channel (use when applicable):

Front A
Back B
Both AB

DD: Day i.e. 01, 02, 03,

MM: Month i.e. 01, 02, 03,

SS: Sequential number 01, 02, 03,

T: Matrix Type (if applicable)

Water W
Solid S
Air A
Oil O
Other X

YY: Year i.e. 12 for 2012

APPENDIX F. PREVENTIVE MAINTENANCE REQUIREMENTS

Item	Frequency	Actions/Comments
Gas purifiers (carrier gas & detector gas)	As Needed	Replacement schedule is based on capacity and grade of gases. In general, replace non-indicating traps every 6-12 months or when indicating traps start to change color. Replace indicating traps when indicating material is spent.
Syringes and/or syringe needles	As Needed	Replace syringe if dirt is noticeable in the syringe, if it cannot be cleaned, if the plunger doesn't slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	As Needed	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	As Needed	Replace with liner or with signs of wear.
Inlet septum	As Needed	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.).
Inlet hardware	As Needed	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Column maintenance	As Needed	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Column Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Column Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules		Replace ferrules when changing columns and inlet/detector parts.
FID Jets & collector	As needed	Clean when deposits are present. Replace when they become scratched, bent, or damaged, or when having difficulty lighting FID or keeping flame lit.
Purge/Sample lines	Annually or as needed	Bake out and purge. Clean with organic free water if necessary.
Trap	As needed	Replace when loss of performance.

Item	Frequency	Actions/Comments
PID	As needed	Clean window

APPENDIX G. REVISION HISTORY

STANDARD OPERATING PROCEDURE: 380 Revision: 8, Effective: 1/15/2014

PURGEABLE AROMATICS AND HYDROCARBONS BY GC PID/FID

Revision	Effective Date	Description
6	06/26/09	 Revised to remove reference to the analysis of BTEX and MTBE and to comply with current SOP format and recent changes to internal COC requirements. Minor edits throughout.
7	03/01/10	 Added the use of 2-methylpentane and 1,2,4-trimethylbenzene as the window defining standard for the integration of gasoline. Added the use of Tekmar/Archon autosampler. Updated to reflect 8015C requirements.
8	1/15/2014	 Changed surrogate spiking procedure to be consistent with EPA Region 9 Laboratory SOP 305. Changed retention time window to default to be in line with other GC procedures. Minor edits throughout.